

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Semi-Annual Progress Report

NGL 33-001-001

Submitted by

ADELPHI UNIVERSITY

March 1969

FACILITY FORM 502

N69-73177	
(ACCESSION NUMBER)	(THRU)
15	None
(PAGES)	(CODE)
CR# 101413	(CATEGORY)
(NASA CR OR TMX OR AD NUMBER)	

Table of Contents

Title	1
Table of Contents	ii
Summary of Progress	1
Reports of Investigators	
Synthesis and Physical Characterization of High Temperature Polymers of Cyanuric Chloride and Siloxanes by F. A. Bettelheim, Principal Investigator R. P. D'Amelia, Research Assistant	3
Magnetic Properties of Type II Superconductors by R. W. Genberg	4
Absorption and Emission Bands of KCl:Tl by Anthony Lemos	9
Thermally Stable Polymers Containing Adamantane Derivatives as Recurring Units by S. Moon and A. Schwartz	10
Aeropaleynological Survey in the Lower Atmosphere in the Mid-Pacific Region by Leslie A. Sirkin	12
X-Ray Investigations of Radiation Damage of Crystals by Alfred Zajac	13

National Aeronautics and Space Administration

Semi-Annual Progress Report
NGL 33-001-001

September 1, 1968 - February 28, 1969

SUMMARY OF PROGRESS

Dr. Genberg reports here on torque measurements on crystalline specimens of vanadium and niobium. Variation of torque with temperature and magnetic field are explained by pinning of the fluxoid structure and Dr. Genberg's results indicate that pinning mechanism is proportional to $1-t^2$ where t is the reduced temperature.

Dr. Lemos' studies of the absorption and emission bands of the alkali halides has been extended to include temperature dependence. The newly predicted transitions appear to agree quite well with experimental data.

Dr. Zajac's progress in the past half year have centered primarily on the reconstruction of experimental apparatus for taking X-ray topographs.

Dr. Bettelheim in conjunction with a graduate assistant, Mr. R. P. D'Amella, reports here on further experimental work on high temperature polymers and specifically on, 1) the thermal and mechanical properties of poly (S-triazine methyl siloxane) and 2) the variations of side chains on the Si atom.

Dr. Sung Moon in conjunction with a graduate assistant, A. Schwartz, reports here on further investigations on thermally stable polymers containing adamantane derivatives as recurring units. Specifically he reports on the synthesis

of what is believed to be polybenzimidazole and 1,3-adamantane dinitrate and a new synthesis for 1,3-dibromoadamantane.

Dr. Leslie Sirkin reports here on progress being made in setting up an aeropalynological survey in the lower atmosphere in the Mid-Pacific region.

TITLE: Synthesis and Physical Characterization of High
Temperature Polymers of Cyanuric Chloride and
Siloxanes

F. A. Bettelheim, Principal Investigator
Mr. R. P. D'Amelia, Research Assistant

At the present time we are pursuing two courses of action:

- 1) The thermal and mechanical properties of our poly(s-triazine methyl siloxane).
- 2) The variations of side chains on the Si atom.

Some of our preliminary results show that the inclusion of the triazine nuclei into a siloxane backbone gives it more high temperature stability than the ordinary methyl siloxane. Most of our products are insoluble and infusible, so that dilute solution studies offer no technique for direct molecular characterization. The mechanical properties are studied via direct reading viscoelastometer. As yet no conclusions can be made regarding the rheological properties of poly(s-triazine methyl siloxane).

. TITLE: Magnetic Properties of Type II Superconductors

R. W. Genberg

Introduction

Until recently investigations in performing magnetization measurements on type II superconductors have assumed that the magnetization was parallel to the applied field. A representative plot of their measurements, including hysteretic effects for increasing and decreasing fields, is shown in Fig. 1; these measurements were conducted using either a ballistic galvanometer or a vibrating sample magnetometer.

Torque Measurements

By using a continuous recording, null-deflection torque balance, we have determined that the torque on single crystalline specimens of superconducting vanadium and niobium not only changes in magnitude with increasing magnetic field but may change sign as well. A representative plot of the torque as a function of increasing field is shown in Fig. 2; for the samples studied hysteretic effects were present as evidenced by the difference between the curves for increasing and decreasing fields.

We recorded the torque along the axis of the cylindrical specimens with the magnetic field being applied in a plane perpendicular to the cylindrical (and crystalline) axis, hence we determined M_{\perp} , the component of the magnetization perpendicular to both the applied field and the cylinder axis. This component was studied as a function of the magnitude of the applied field, with the orientation of the field relative to the crystalline axis and the temperature of the specimen as controlled parameters.

We have found in vanadium and niobium that: (1) the torque exists only in the superconducting state and it varies in a reproducible manner with the magnitude of the applied field, (2) the variation of the torque with applied field is orientation dependent, (3) the torque decreases with increasing

temperature and vanishes at T_c , and (4) the torque is not generated as a result of the time rate of change of the applied field.

Presently the existence, hence variation, of the torque is not accounted for in the theory of type II superconductors. Assuming that the torque arises from pinning - with a resultant bending - of the fluxoid structure within the superconducting specimen, we have been able to make a preliminary determination of the temperature dependence of the pinning mechanism. If we write the torque as

$$\mathcal{T} = 4\pi \mu f(t) MH \quad (1)$$

then as $H \rightarrow H_{c2}$ we have

$$\mathcal{T} = f(t) \frac{H(H - H_{c2})}{2K_2 - 1} \quad (2)$$

where we have utilized the Abrikosov expression for the magnetization for H near H_{c2} , i. e.,

$$M = \frac{H - H_{c2}}{(2K_2 - 1)}^2 \quad (3)$$

Since $f(t)$ includes the fraction of pinned fluxoids, it is related to the strength of the pinning mechanism.

From equation (2) a plot of \mathcal{T}/H as a function of H at fixed t should yield a straight line whose intercept is H_{c2} and slope is $f(t)/(2K_2 - 1)$. From our data and a determination of K_2 - estimated for our samples using the data of French⁽¹⁾ and the calculation of Ellenberger⁽²⁾ - preliminary results indicate that the temperature dependence of the pinning mechanism is proportional to $1-t^2$, where t is the reduced temperature.

To establish this more precisely we are presently attempting to measure $M_{||}$, the parallel component of the magnetization, in order to determine accurately K_2 for our specimens. This should then result in a precise determination of the temperature dependence of the pinning interaction.

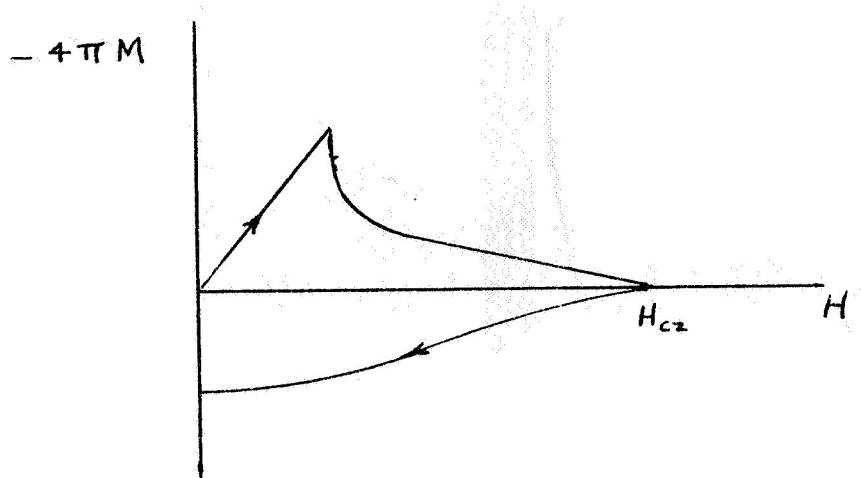
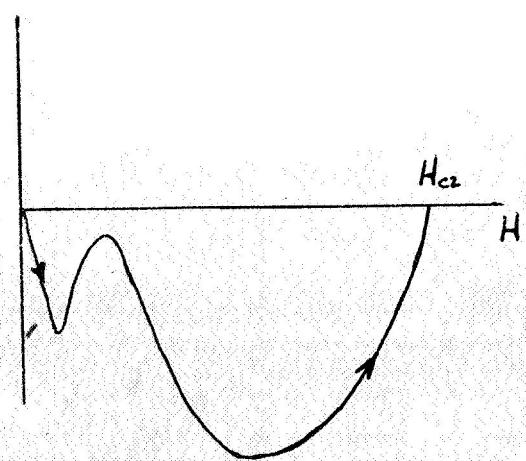
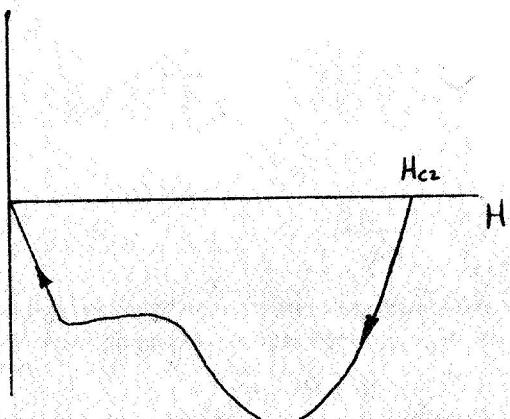


Fig. 1



(a)



(b)

Fig. 2

References

- (1) R. A. French, Physics Letters, 23, 59 (1966)
- (2) G. Ellerberger, Phys. Rev. 153, 584-98 (1967)

Captions:

Fig. 1 A representative plot of the component of the magnetization parallel to the applied field.

Fig. 2 A representative plot of the torque for increasing and decreasing fields.

TITLE: Absorption and Emission Bands of KCl:Tl

Anthony Lemos

The theoretical investigation of the optical absorption bands of KCl:Tl has been extended to include temperature dependent variations in the band structure. The structure of the bands arises from the electron-phonon interaction. Such an interaction gives rise to temperature dependent splitting of certain energy levels, which in turn gives rise to new transitions. The results of our calculations agree very well with experiment. Recently we have published two abstracts concerning this work in the Bull. Am. Phys. Soc. 14, (1969). A paper is being prepared at the present time and should be submitted for publication within a month.

Two new related problems are now being investigated. These are 1) the emission spectra associated with KCl:Tl and 2) the F-center life times. We feel confident that using the techniques developed during the absorption work, these two problems can be solved.

TITLE: Thermally Stable Polymers Containing Adamantane Derivatives as Recurring Units

S. Moon and A. Schwartz

In our program of preparing heat resistant polymers containing the adamantane nucleus as a repeating unit, we wish to report that, with the aid of Dr. J. Hecht of Dow Chemical Co. (Mass.), a heat resistant polymer has been synthesized. This material is produced by heating together diphenyl 1,3-adamantane dicarboxylate and diamino benzidine at 250° C for two hours and at 280° for an additional hour. This material is believed to be a polybenzimidazole.

Thermal analysis of this material shows that it is indeed quite heat resistant. Only at a temperature of 870° did the polymer start to show a very gradual decomposition. Heating at 1000° for 2 hours resulted in considerable charring but most of the material could still be recovered in solid form.

The hitherto unknown 1,3-adamantane dinitrate also has been produced from the corresponding dibromide. Heating this material at 190° for one hour in a sealed tube with tetrachloroethylene resulted in a considerable amount of what appears to be good polymeric material. The properties of this material are being investigated. Also, from this same reaction mixture were isolated some unknown ketones. On the other hand, heating the dinitrate in a hydrogen donating solvent such as decane resulted mainly in the production of 1,3-adamantanediol. In this case little polymer was produced.

A useful and reproducible synthesis for preparing the difficult to synthesize 1,3-dibromoadamantane has been accomplished. The dibromo compound is the basic unit from which all the other adamantane derivatives used as polymerization monomers are made. Without its facile synthesis the polymer program would be severely hampered.

However, work has been begun on the synthesis of the unknown adamantane isocyanide dichloride. This material will be prepared from the available isothiocyanate and a method will be developed for producing polymers from the more readily available mono adamantane compounds.

* TITLE: Aeropalynological Survey in the Lower Atmosphere
In the Mid-Pacific Region

Leslie A. Sirkin

Samples collected by Dr. Holzapfel in flights over the Mid-Pacific region during the summer of 1968 have been provided and are being analyzed at the present time. Dr. Sirkin met with Dr. Holzapfel when the aircraft was brought to New York for servicing and for equipment modification in the Fall of 1968. Dr. Holzapfel instructed Dr. Sirkin and other interested scientists who are working in atmospheric particle collecting, in the operation of the aircraft sampling device on board the aircraft, at the aircraft company's facility on Long Island. Additional samples for analysis will be available early in 1969 when the aircraft is again operational.

Dr. Sirkin is negotiating with botanists from the Bishop Museum in Honolulu for reference materials and pollen reference slide collections from the Mid-Pacific region in order to facilitate study and analysis of the material obtained from the atmosphere. It has not been decided whether the Museum will be able to provide this material or whether Dr. Sirkin will have to go to Honolulu to collect it himself at the Museum.

Lastly, Dr. Holzapfel had indicated that sampling during 1969 will also be conducted on board an ocean going vessel in order to obtain atmospheric samples near the ocean surface.

TITLE: Aeropalynological Survey at Wallops Island, Virginia

Leslie A. Sirkin

Manuscript for this study is in progress and should be ready to submit for publication in the Spring of 1969. Data reduction and drafting of the major diagram was concluded late in 1968 so that conclusion of the manuscript in the Spring of 1969 is feasible.

TITLE: X-Ray Investigations of Radiation Damage of Crystals -

Alfred Zajac

During the Fall 1968 semester, most of the time was spent in redesigning and constructing the experimental arrangement for taking x-ray topographs. The method of taking x-ray topographs consists in reflecting an x-ray beam from Bragg planes of the chosen crystal, with the beam going through the crystal plate; the crystal is slowly translated along its length, and at the same time it oscillates about the Bragg angle. We have completed the construction of the apparatus; its unique feature is an x-ray collimator which is five feet long. We have obtained x-ray topographs from perfect silicon crystals.

We are not satisfied with our design yet, and will proceed with some further modifications.